Indian Journal of Chemistry
Vol. 37B, November 1998, pp. 1219-1220

Note

Isolation and characterisation of a novel amide from *Michelia champaka*

Sanjay Sharma & B K Mehta

Natural Product Laboratory, School of Studies in Chemistry, Vikram University, Ujjain- 456001, India

Received 11 June 1997; accepted (revised) 11 September 1998

The benzene extract of *M. champaka* (anthers) yields a new compound *N*-methyl (3-ene-15-hydroxy pentadecanyl) amide. It has been characterized on the basis of spectral data (IR, PMR and Mass).

Michelia champaka belongs to family *Magnoliaceae* and has been described as a medicinally important plant in the indigenous system of medicine[1,2]. The anthers of its flowers are reported to prevent pregnancy[3,4] and the plant is used as a contraceptive. From its trunk bark some active principles were reported by Majumdar and coworkers[5]. Sethi *et al.*[7,8] reported a new sesquiterpene lactone micheliolide with parthenolide, dihydroparthenolide and consunolide were reported from the roots of *M.champaka*. Wang has studied the essential oil obtained from *M. alba*[9]. Yang and workers reported alkaloids ushinsumine, oxoushinsumine, michelabine and an unknown base from the plant of *M. champaka*[10].

In the present communication we describe the isolation of a novel amide. Separation of benzene extract of anthers on silica gel column yielded a white crystalline compound. Recrystallization from methanol in refrigerator for one month yielded pure compound **1.** The mass spectrum and elemental analysis revealed its molecular formula to be $C_{17}H_{33}NO_2$. The IR spectrum showed bands at 2960, 2940, 2860,1480, 1440 and 1420 cm^{-1} revealing its paraffinic nature. A band at 3400 cm^{-1} showed the presence of a hydroxyl group. Bands at 3200 and 1260 cm^{-1} showed the presence of a secondary amino group. A band at 1650 cm^{-1} showed the presence of a double bond and keto group of amide. The ^1H NMR spectrum showed doublet at δ 2.3 for three protons due to the methyl protons attached to the nitrogen of the amide. A multiplet at δ 3.02 for six protons was assigned to three methylenes directly attached to carbonyl and double bond. The methylene protons attached to the OH group resonated at δ 2.3 as a multiplet. All other methylene protons resonated at δ 1.66. The olefinic protons gave a multiplet at δ 5.96. The hydroxyl proton resonated at δ 1.98. The amino group resonated at δ 6.62[11-13]. The mass spectrum showed it to be a straight chain compound (base peak at m/z 55). The α-cleavage produced fragments at m/z 55 and 58 confirming the position of amide group and double bond in the molecule. Formation of fragments at m/z 84 and 170 confirmed the position of double bond at C-3[14].

On the basis of the above data compound **1** was characterized as *N*-methyl-(3-ene-15-hydroxy- pentadecanyl)amide. This is a new compound and being reported for the first time by us.

1

Experimental Section

The melting point is uncorrected. IR spectrum was recorded on Perkin Elmer 377-B in KBr, ^1H NMR spectrum in $CDCl_3$ with TMS as internal standard, on Brucker WM 300 MHz instrument and Mass spectrum on Jeol JMS D –300 mass spectrometer. Silica gel was used for column chromatography. The anthers of *M. champaka* were collected from Ujjain city and its nearby area. The identification of the material was confirmed by the authorities of School of Studies in Botany, Vikram University, Ujjain.

Extraction and Isolation. The shade dried and powdered anthers of *M. champaka* were extracted with benzene for 64 hr. Solvent was removed by rotary film evaporator. The resultant solid mass was put on a silica gel column for separation. The column was eluted with different solvents in the increasing order of polarity. The fractions of similar compositions were mixed and solvent was removed.

Identification of the compound. It was isolated from benzene : ether fraction of the column and recrystallized from methanol, m.p. 52°C. Anal.

Found : C, 72.84; H, 11.52; N, 5.03. Calcd for $C_{17}H_{33}NO_2$: C, 72.66; H, 11.66; N, 4.95 %. It was identified as *N*-methyl-(3-ene–15-hydroxy pentadecanyl) amide, on the basis of the following spectral data.

PMR (CDCl $_3$): δ 1.66 (s,18H, 9-CH_2) 1.98 (s, 1H, OH) 2.3 (d, 5H, N-CH_3–OH-CH_2, *J*=5 Hz) 3.02 (m, 6H, 3-CH_2, *J*=5 Hz) 5.96 (m, 2H-CH=CH, *J*=5 Hz) and 6.62 (bs,1H, N-H); MS: m/z 283 (10), 255 (30.4), 239 (15.2), 212 (15.0), 202 (8.3), 198 (15.0), 185(25.0), 170 (20.0), 166 (10.5), 155 (15.0), 113 (40.3), 84 (55), 60 (38) and 55 (100).

Acknowledgement

We thank ICMR, New Delhi for the award of Junior Research Fellowship to Sanjay Sharma. Thanks are also due to RSIC, CDRI, Lucknow for mass and ^{1}H NMR spectra and elemental analysis.

References

1 Koman M C, *Reports on the investigation of indigenous drugs,* 3 rd Rep, **1920**, 6.
2 Mhaskar K S & Caius J F, *Indian Med Res Mem*, 19, **1931**, 54.
3 Shastri N I, *Bhavprakash nighantu* (Ramkumar Press, Lucknow), **1964**, 29.
4 Billore K V & Audichya K C, *J Res Ind Med Yoga Homeop*, 13, **1978**, 104.
5 Majumdar P L & Chatterjee A, *J Indian Chem Soc*, 40, **1963**, 929.
6 Wellburn A R, Stevenson J H, Hemming F W & Morton R A, *Biochem J,* 102, **1967**, 3137.
7 Sethi V K, Dhar K L & Atal C K, *Planta Medica*, 51, **1984**, 364.
8 Sethi V K , Dhar K L & Atal C K, *Indian Pharm Sci*, 40, **1978**, 226.
9 Wang D J, *Nat Sci Coun Mon*, 7, **1979**, 1034.
10 Yang T H & Hsiao C Y, *J Pharm Soc, Japan*, 83, **1963**, 243
11 Bellamy L J, *Infra red spectra of complex molecules* (Chapman & Hall, London), **1975**, 231-240.
12 Dyer J R, *Applications of absorption spectroscopy of organic compounds* (Prentice Hall of India Pvt. Ltd., New Delhi), **1984**, 86-95.
13 Jackman L M & Sternhell S, *Applications of nuclear magnetic spectroscopy in organic chemistry* (Pergamon Press, New York), **1969**, 72.
14 Silverstein R M, Bassler G C & Morril T C, *Spectrometric identification of organic compounds* (John Wiley, New York), **1981**, 25.